| ANALYST: | | VPDES NO | |
|---|---|---|---|

Parameter: Oil & Grease
Method: HEM
05/04

METHOD OF ANALYSIS:

| | |
|---|---|
| | EPA Method 1664 Revision A |

| | | Y | N |
|---|---|---|---|
| | **COLLECTION** | | |
| 1) | Are glass bottles with Teflon (PTFE) lined screw caps used for sample collection? [6.1.1] | | |
| 2) | Have collection bottles been properly cleaned? NOTE: If blanks demonstrate that the collection containers and lids are clean, the cleaning preparation may be eliminated. (Bottles - detergent washed, rinsed with water, and then either solvent rinsed or baked at 200-250EC for 1 hr prior to use? Liners for screw caps - detergent washed, rinsed with water and solvent rinsed and baked at 110-200EC for 1 hr prior to use?) [6.1.2] | | |
| 3) | Is enough headspace left at top of collection bottle to allow for pouring without loss of sample? | | |
| 4) | Are samples adjusted to a pH of <2 with HCl or $H_2SO_4$ and refrigerated at 0-4EC? [8.1.1 & 8.4] | | |
| 5) | Are samples analyzed within 28 days of the date and time of collection? (8.5) | | |
| | **QUALITY CONTROL** | | |
| 6) | Have MDLs been determined? [9.2.1] | | |
| 7) | Has initial precision and recovery been demonstrated? [Analyze 4 precision and recovery (PAR) standards and compute average percent recovery (X) and standard deviation of % recovery (s). Results must meet Acceptance Criteria -- HEM (s) #11; (X) 83-101 [9.2.2] | | |
| 8) | Are matrix spikes performed on 5% of samples from a given discharge/waste stream by adding the spiking solution to the sample container? [9.3] | | |
| 9) | Are spike recoveries within the acceptable range? HEM - 78-114% [9.3.4] | | |
| 10) | Are laboratory reagent water blanks analyzed to demonstrate freedom from contamination? [9.4] | | |
| 11) | Is balance calibration verified using Type I (formerly Class S) weights at beginning and end of each day of analysis? NOTE: Calibration must be within 10% at 2 mg and 0.5% at 1000 mg. [9.5 & 10.0] | | |
| 12) | Is a PAR standard which is added to a sample container, analyzed with each batch? Results must meet Acceptance Criteria -- HEM (X) 78-114% [9.6] | | |
| 13) | Is level of standard marked on container after each day of use and then reconstituted with acetone prior to subsequent use? [7.10.2 & 7.10.3] | | |
| 14) | Is a quality control sample (QCS), from source different than the standard used, routinely analyzed? [9.7] | | |
| | **PROCEDURE** | | |
| 15) | Are samples brought to room temperature prior to analyses? [11.1.1] | | |
| 16) | Is sample volume determined by either marking water meniscus or weighing bottle for later determination? [11.1.4] | | |
| 17) | Is pH of <2 verified by dipping a glass rod into sample; touching rod on pH paper; then rinsing rod with hexane over the sample, thus including the rinsate in the sample extraction? [11.2.1] | | |
| 18) | Is all glassware solvent rinsed or baked at 105-115EC after cleaning? [4.3] | | |

| | | Y | N |
|---|---|---|---|
| 19) | Are cleaned boiling flasks containing 3-5 boiling chips baked at 105-115EC for a minimum of 2 hrs. prior to being placed in desiccator for cooling and determining the tare weight? [11.3.1] | | |
| | **Separatory Funnel Extraction** | | |
| 20) | Are 30 mLs of n-hexane added to sample bottle, which is then sealed with original cap; shaken to rinse all interior surfaces; and then poured into the separatory funnel ( 2 L funnel fitted with a Teflon stopcock)? [11.3.3 - 6.4.3] | | |
| 21) | Is sample extracted by vigorously shaking the separatory funnel for 2 mins.? [11.3.4] | | |
| 22) | Are layers allowed to separate for a minimum of 10 mins. before draining the lower layer into the original sample container? [11.3.5 & 11.3.6] | | |
| 23) | Is the tared weight of the distilling flask recorded? [Permit] | | |
| 24) | Is solvent layer drained through a Whatman 40 (or equivalent) filter holding approximately 10 g of pre-rinsed sodium sulfate ($NaSO_4$) into a tared boiling flask? [11.3.8] | | |
| 25) | Are 3 extractions performed on each sample? [11.3.9] | | |
| 26) | Is a small amount of n-hexane drained from separatory funnel with each extraction? [11.3.6] | | |
| 27) | Are the separatory funnel tip, filter paper, and funnel rinsed with 2-3 small portions of n-hexane which is then added to the flask? [11.3.10] | | |
| | **Solid Phase Extraction** | | |
| 28) | Is there a SOP available for SPE? [Permit] | | |
| 29) | Is sample bottle rinsed several times using n-hexane with rinsate being added to the filter? [11.3.3] | | |
| 30) | Is filter kept moist from time of conditioning until after sample is filtered? [Permit] | | |
| 31) | Elution<br>a) Is sample eluted with several aliquots of n-hexane? [11.3] | | |
| | b) Is a small amount of each n-hexane rinse pulled through filter with remaining solvent held in filter for a minimum of 2 mins. prior to starting next rinse? [Permit] | | |
| | c) Are sides of reservoir above filter rinsed with n-hexane? [Permit] | | |
| 32) | Are eluents passed through approximately 10 g of pre-rinsed sodium sulfate ($NaSO_4$) into a tared boiling flask with collection vessel rinsate added to flask also? [11.3.8] | | |
| | **Evaporation** | | |
| 33) | Is solvent collected during the evaporation process for reuse? [11.4.1] | | |
| 34) | Is temperature of water bath or steam bath adjusted to allow concentration to be completed in 30 mins.? Approx. 85EC [11.4.1] | | |
| 35) | Is the sample allowed to distill until the flask appears dry or distillation head reaches 70EC? [11.4.2] | | |
| 36) | Following evaporation, is the flask swept of solvent fumes with a vacuum for 15 secs.? [11.4.2] | | |
| 37) | Is the flask wiped clean of moisture and fingerprints, dried in oven at 70 " 2EC for 30 min., then placed in a desiccator for 30 mins. minimum prior to weighing? [11.4.2 - 11.4.4] | | |
| 38) | Was drying cycle repeated until weight loss was < 4% of previous wt. or < 0.5 mg, whichever was less? [11.4.4] | | |
| 39) | Is weight of the distilling flask and residue recorded? [11.4.4] | | |

| | Y | N |
|---|---|---|
| 40) Is weight of the residue calculated and recorded? [11.4.4.1] | | |
| 41) Is the volume of the original sample determined and recorded? [11.4.5] | | |
| 42) Is calculation for the concentration of HEM (oil and grease) correct and shown on the bench sheet? [12.1] | | |

$$HEM(mg/L) = \frac{W_h(mg)}{V_s(L)}$$

where :
$W_h$ = Weight of extractable material
$V_s$ = Sample volume

PROBLEMS: